(2.94 g, 1.05 eq. based on $MeSO_3H$) and concentrated in vacuo. To the resulting residue was added isopropanol (120 mL) and the mixture was concentrated in vacuo to give 88 g of a residue.

The residue was dissolved in isopropanol (37 g) at 47° C. The resulting solution was cooled down to 2° C. during 2.5 h; crystallization started spontaneously at 30° C. The crystalline product was isolated by filtration, washed with isopropanol (3×20 mL, 0° C.) and dried in vacuo (17 h at 35° C.) to give 10.1 g of a white crystalline product which according to GC consisted of 96.4 wt % α-(4) and 0.065 wt % β-(4), corresponding with a total yield based on E-R-3-(2,2-dimethyl-[1,3]dioxolan-4-yl)-acrylic acid ethylester of 9% and an α-(4): β-(4) ratio of >1000:1.

Thus, the total yield of the first and second crop of α-(4) based on E-R-3-(2,2-dimethyl-[1,3]dioxolan-4-yl)-acrylic acid ethylester was 46%.

## Example 8

### Preparation of pure (3R,3aS,6aR) hexahydro-furo[2,3-b]furan-3-ol from α-(4) intermediate

The procedure described in WO03/022853, Example IV, last step, was followed.

## Example 9

### Preparation of pure α-(4) from R-3-(2,2-dimethyl-[1,3]dioxolan-4-yl)-acrylic acid ethylester by Direct Crystallization of α-(4) from a Crude Mixture of β-(4) and α-(4) and Simultaneous Epimerization of β-(4) to α-(4)

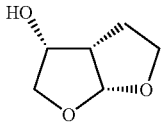
To R-3-(2,2-dimethyl-[1,3]dioxolan-4-yl)-acrylic acid ethylester (399.5 g, 75.1 wt % pure, 1.5 mol) was added nitromethane (915.0 g of a 11 wt % solution in methanol, 1.65 mol, 1.1 eq.) and the solution was cooled to 0° C. Subsequently, DBU (233.3 g, 1.5 mol, 1 eq.) was added dropwise during 50 min at 0-5° C. and the reaction mixture was heated up to 20° C. and stirred for another 16 h at that temperature. The resulting reaction mixture was cooled to 0° C. and NaOMe (594.0 g of a 15 wt % solution in methanol, 1.65 mol, 1.1 eq.) was added dropwise during 50 min at 0° C. The resulting solution was stirred for 1 h at 0° C. and quenched into a solution of $H_2SO_4$ (368 g, 96 wt %, 3.6 mol, 2.4 eq.) in methanol (370 g) at 0-5° C. by dropwise addition during 3 h under vigorous stirring. The reaction mixture was stirred for 2 h at 0-5° C. and then quenched into a stirred slurry of $KHCO_3$ (457.6 g), in water (870 mL) at 0-5° C. by dropwise addition during 1 h. $KHCO_3$ was portionwise added to keep the pH above 3.5. The formed salts were removed by filtration at 0-5° C. and washed with methanol (530 mL). After concentration in vacuo of the combined filtrate and washing to approximately 1000 ml the aqueous phase was extracted with toluene (2×2100 mL, 3×1050 mL). The combined organic phases were concentrated in vacuo, giving 202.9 g of a semi-solid.

Subsequently, methanol (42.6 g) and $MeSO_3H$ (6.06 g, 0.04 eq.) were added and the mixture was heated up to 50° C. After 2 h of stirring at this temperature the mixture was cooled to 20° C. and stirring was continued for an additional 12 h. After cooling to −5° C., triethyl amine (6.60 g, 1.1 eq. based on $MeSO_3H$) was added and the mixture was stirred for another 2 h. The crystalline α-(4) which was isolated by filtration, washed with cold (−5° C.) isopropanol (3×70 mL) and dried on the air. This gave 120.0 g α-(4) which, according to quantitative GC analysis, was 99.0 wt % pure and contained 0.09 area % of β-(4). This corresponds to a yield of 51% based on R-3-(2,2-dimethyl-[1,3]dioxolan-4-yl)-acrylic acid ethylester.

The invention claimed is:

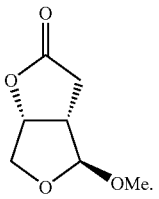
**1**. A method for the synthesis of (3R,3aS,6aR) hexahydro-furo[2,3-b]furan-3-ol having the structure of formula (6),

(6)




which method comprises the step of reducing the intermediate of formula α-(4):

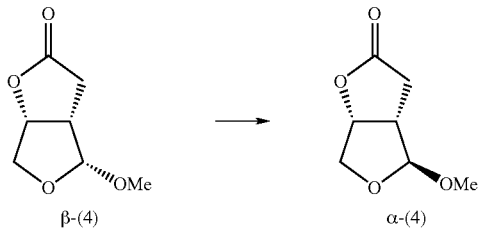
α-(4)




**2**. A method according to claim **1** which method further comprises crystallizing intermediate of formula α-(4) with a solvent prior to the reduction thereof.

**3**. A method according to claim **1** which method further comprises

a) epimerizing with acid intermediate of formula β-(4) into the intermediate of formula α-(4); and




b) crystallizing intermediate of formula α-(4) with a solvent prior to the reduction thereof.

**4**. A method according to claim **3** which method further comprises after crystallizing intermediate of formula α-(4),

a) epimerizing with acid intermediate of formula β-(4) in the mother liquor of said crystallization into the intermediate of formula α-(4); and